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EXECUTIVE SUMMARY

DETERMINATION OF RADIUM-226 IN FLYASH LEACHATES AND GROUND WATERS

S. P. Sood and F.R. Greening

Analytical Services Section

In support of Ontario Hydro's program of monitoring existing and proposed ash disposal sites for their impact on the environment, a method for the determination of radium-226 in samples of ground water and flyash leachates has been developed. A minimum detectable activity limit of 0.2 pCi/L was established which is well below the target concentration in drinking quality water of 3 pCi/L set by the Ministry of the Environment of Ontario. Several samples of ground and surface water from Domtar and Booth quarries and the proposed Atikokan site have been analyzed. The radium-226 content in most samples was found to be less than 1 pCi/L.

job	file	date	report no.
740633-663-000 740633-501-000	830.6148	February 20, 1985	85-35-K

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To Mr. F.J. Kee
Director of Research

DETERMINATION OF RADIUM-226 IN FLYASH LEACHATES AND GROUND WATERS

S.P. Sood and F.R. Greening

A method for the determination of radium-226 in samples of ground water and flyash leachate based on its separation on a specially prepared barium sulphate suspension followed by alpha spectrometry has been developed. The accuracy of the method was checked by using a low level Environmental Protection Agency cross-check sample of known value. The detection limit of the procedure has been evaluated to be 0.2 pCi/L which is well below the target concentration in drinking quality water of 3 pCi/L set by the Ontario Ministry of the Environment. A large number of ground water samples and flyash leachates have been analyzed. The chemical recovery of radium as determined with a barium-133 tracer was found to be 85 +/- 10%. The radium content in these samples was generally less than 1 pCi/L. The counting statistics accounted for most of the uncertainty in the determination.

1.0 INTRODUCTION

The generation of electricity by coal-fired power stations can result in the release of trace elements to the aquatic environment through leaching from flyash at ash disposal sites. Ontario Hydro measures these emissions through a project contracted to the Chemical Research Department by Design and Development Division/1-3/. Increased awareness throughout the world of the radiological impact associated with the generation of electricity from coal has resulted in several studies on the radionuclide content of coals and ashes to evaluate the extent to which these can enter the environment/4-8/.

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The radon-222 emanation from samples of Lakeview GS flyash has recently been studied at Chemical Research/8/ and the present report describes the development of a rapid and sensitive low level analysis method for the determination of radium-226 in flyash leachate and ground water samples from ash disposal sites.

2.0 RADIONUCLIDES IN SURFACE AND GROUND WATERS

Naturally occurring radionuclides (e.g. potassium-40, carbon-14, tritium and members of the thorium-232, uranium-235 and uranium-238 series) contribute to the radioactivity in the aquatic environment. The levels at which these radionuclides are present in the environment varies from place to place and from time to time. In general less than a few pico curies of alpha activity per liter of water are present. However, industrial activity (e.g. mining and processing of minerals containing radionuclides and the generation of power) may alter these levels. In Ontario the Ministry of the Environment (MOE) therefore regulates the discharges from various industrial sources through Certificates of Approval, guidelines for water management/9/ and drinking water quality objectives/10/.

For radium-226, the MOE has set the Maximum Acceptable Concentration (MAC) in drinking water to be one percent of the International Commission on Radiological Protection (ICRP) recommended annual occupational dose equivalent limit for continuous exposures (i.e. 1 Becquerel per liter (27 pCi/L)). It has also set a Target Concentration (TC) of 0.1 Becquerel per liter (3 pCi/L) which is one tenth of the MAC based on a life long continuous consumption/10/.

Ontario Hydro has a program to monitor the surface and ground water quality at existing ash disposal sites / 1,2 / and carries out pre-operational surveys at proposed disposal sites/3/. In order to meet these program objectives, development of a method for the determination of radium-226 at the TC level was initiated.

3.0 DETERMINATION OF RADIUM IN WATER

The Standard Method for the determination of radium in water is its co-precipitation as radium sulphate in the presence of barium and lead carriers followed by the purification of barium (radium) sulphate. The sulphates are counted by a gas flow proportional counter or scintillation counter/11/. This method is quite slow as it involves two slow sulphate precipitation steps. It has recently been shown that a specially prepared barium sulphate suspension can be used to rapidly collect sulphates which can co-precipitate on it /12/. The method described in this report uses the co-precipitation of radium with 50 micrograms of barium as carrier onto a specially prepared barium sulphate suspension. Complexing agents are used to prevent carry over of other alpha emitting elements. The precipitation of barium sulphate at trace concentrations is very slow and needs

seeding to initiate the process. However, in the presence of the specially prepared barium sulphate suspension the barium and radium in the solution quickly deposit as a thin layer on the activated surface of the added suspension . In addition, the resultant precipitate can easily be filtered. The chemical recovery of the procedure is measured with a barium-133 tracer. The alpha radiation from radium does not suffer much absorption losses because it traverses a very thin layer of freshly precipitated barium sulphate and as a result, radium-226 can be determined quantitatively by alpha spectrometry.

4.0 EXPERIMENTAL PROCEDURE

4.1 Instrumentation

All alpha activity measurements were made with an EG&G ORTEC Model 576 alpha spectrometer equipped with two 450 mm^2 silicon surface barrier detectors and coupled to an EG&G ORTEC Model 7052 Data Acquisition and Analysis System. Gamma activity of the barium-133 tracer was measured using a 76mm X 76mm NaI(Tl) detector coupled to a Canberra Series 30 multichannel analyzer.

4.2 Summary Description of Procedure

4.2.1 Preparation of Barium Sulphate Suspension

Barium nitrate, 0.5 g, was dissolved in 60 mL of distilled water in a 250 mL Erlenmeyer flask. Anhydrous potassium sulphate, 24 g, and concentrated sulphuric acid, 13 mL, were added and the resultant slurry evaporated until the barium sulphate formed redissolved. The flask was cooled and the residual cake was slurried in 150 mL of distilled water with heating. The resulting suspension was cooled and the volume made up to 200 mL. The barium sulphate precipitate was resuspended by shaking before use.

4.2.2 Optimization of the Addition of Barium Sulphate Suspension

A standard solution containing 5.9 micro curies of radium-226 (Amersham Cat. No RAY.44) was diluted to 1 liter with 0.5 mol/L nitric acid. A small diameter source was prepared by evaporating 50 µL of this solution on a planchet which was then counted.

Approximately 6 micro curies of barium-133 was diluted to 100 mL with 0.5 mol/L HCl. An aliquot (100 µL) of the above solution was deposited on a 47 mm diameter Millipore filter contained in a plastic petridish and evaporated and sealed. The filter was counted in exactly the same manner as the samples each time to evaluate the recovery of the precipitation step.

To a liter of distilled water samples acidified with 5 mL of concentrated HCl and containing 50 µL of radium-226 solution, 100 µL of barium-133 tracer solution and 100 µL of barium solution (0.5 g Ba/L) were added. Then 1, 3, 5 or 10 mL of the barium sulphate suspension was added to co-precipitate barium (radium) sulphate. The precipitate was digested for one hour at approximately 90 ^{o} C, cooled and then filtered through a 0.45 µm Millipore filter and counted both for barium-133 activity and alpha activity. The chemical recovery was obtained from the sample count and the standard source count. The relative alpha activity was evaluated as the ratio of the sample activity to the standard point source activity. The results obtained are shown in Figure 1 which shows that as the amount of added suspension increases, the chemical recovery improves. The relative alpha activity also increases initially due to improved chemical recovery. However, as the quantity of suspension added becomes large, the measured alpha activity decreases due to the increased thickness of the source. The optimum amount of the suspension was found to be about 5 mL.

4.3 Counting Efficiency of Alpha Detectors

Three sets of duplicate samples of standard radium-226 solution were diluted to one liter with dilute HCl (5 % by volume). After addition of barium-133 tracer, barium (radium) sulphate was precipitated according to the procedure described in the appendix. The precipitate was filtered and counted for barium-133 activity to evaluate chemical recovery. The spectrum of the emitted alpha particles was recorded on the alpha spectrometer, a typical spectrum being shown in Figure 2. The area below the 4.8 Mev energy peak was integrated to evaluate the radium-226 activity. A reagent blank was prepared in a similar fashion and the alpha activity in the region of interest determined. The net radium activities of the samples were corrected for the chemical recovery and the detector efficiency evaluated to be 9.0 +/- 0.6 %.

5.0 RESULTS AND DISCUSSION

5.1 Determination of Radium in Ground Water Samples

Very little activity of radium-226 was found in all the samples of ground water from Domtar and Booth quarries and the proposed ash disposal site at Atikokan TGS. In general the radium contents were found to be less than 1 pCi/L. The precision of the chemical recovery step as monitored by the barium-133 tracer was good (85 +/- 10 %).

5.2 Estimation of Errors

Radioactive disintegration occurs in a random manner described by the Poisson distribution and the standard deviation of a large number of events, N, equals the square root of N.

$$\sigma (N) = N^{1/2}$$

For a large number of events, N > 20, the Poisson distribution is approximated to a normal (Gaussian) distribution with a mean of N and a standard deviation equal to the square root of N. The overall uncertainty of a measurement is calculated by the usual method for the propagation of errors. For several samples of ground water containing < 1 pCi/L of radium-226, the error due to the counting statistics has been found to be the most important source of uncertainty in the final result.

5.3 Accuracy of the Method

An EPA supplied solution containing radium-226 and radium-228 was analyzed for the radium-226 content. A value of 5 +/- 1 pCi/L was obtained. This compares very well with the reported value of 4.1 +/- 0.6 pCi/L.

5.4 Lower Limit of Detection (LLD)

The LLD is defined as the smallest quantity of sample activity that will result in net counts for which there is 95 % confidence level of certainty that radioactivity is present above the reagent blank. For a reagent blank of N(b) per 1000 minutes the LLD can be evaluated as:

$$LLD = 4.66 \times N(b)^{1/2} / 2.22 \times 1000 \times E \quad pCi$$

where E is the detector efficiency.

The reagent blank has been determined at various times and after each new barium sulphate suspension preparation. The value varies from 20 to 45 counts per 1000 minutes. This results in LLD values of 0.1 to 0.2 pCi. For a one liter sample this will amount to a minimum detectable activity of approximately 0.2 pCi/L. This is well below the target concentration for the drinking quality water of 3 pCi/L established by the MOE.

5.5 Interference from Other Alpha Emitting Radium Isotopes and Decay Products of All Radium Isotopes

Besides radium-226 three other isotopes of radium, namely, radium-228, radium-224 and radium-223 can be present in the natural ground water samples. Decay products of radium-224 (radon-220 and polonium-216) and of radium-223 (radon-219 and polonium-215) enter into equilibrium with the parent nuclei within a few minutes of chemical separation.

Other alpha emitting daughter isotopes (radon-222, polonium-218, polonium-214, polonium-212 and bismuth-211) will grow in the sample with the passage of time resulting in an increase in the measured total alpha activity. The decay characteristics of the different members of the chains with half-lives of less than 30 days are listed in Table 1. It can be seen from the table that all of the other alpha emitting radionuclides have alpha particles of energies greater than that of radium-226. By restricting the integration of the counts in the alpha spectrum to between 3.4 and 4.8 Mev the influence of these radionuclides in the determination of radium-226 has been practically eliminated.

6.0 SUMMARY AND CONCLUSIONS

A method for the separation of radium from aqueous samples based on its co-precipitation with barium as sulphates on a barium sulphate suspension and its subsequent determination by alpha spectrometry has been developed. Good chemical recovery of the radium-barium has been obtained by using an optimized quantity of the suspension. The counting must be performed within a reasonably short period after separation (within a day) as a long delay will increase the amount of alpha emitting daughters within the sample as shown in Figure 3.

Large counting times are required as the sample activities are very low. This fact also necessitates extreme caution in processing samples in order to avoid contamination.

The radium-226 content of samples of flyash leachates and ground waters collected at ash disposal sites was below the MOE target concentration for drinking quality water.

7. ACKNOWLEDGEMENT

Technical assistance of Mr. G. Adams during the development of the method is acknowledged.

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TABLE 1

RADIUM ISOTOPES AND DAUGHTERS WITH HALF-LIFE LESS THAN 30 DAYS

NUCLIDE		RADIATION		Half-life
Parent	Daughter	Type	E-alpha, Mev	
^{226}Ra		Alpha	4.78 (95 %)	1600 years
			4.59 (5 %)	
	^{222}Rn	Alpha	5.48	3.8 days
	^{218}Po	Alpha	6.00	3 min.
	^{214}Pb	Beta		27 min.
	^{214}Bi	Beta		20 min.
	^{214}Po	Alpha	7.68	164 us.
^{223}Ra		Alpha	5.71 (50 %)	11.7 days
			5.60 (24 %)	
			Others	
	^{219}Rn	Alpha	6.81 (82 %)	4 s.
			6.55 (13 %)	
			6.42 (5 %)	
	^{215}Po	Alpha	7.36	0.002 s.
	^{211}Pb	Beta		36 min.
	^{211}Bi	Alpha	6.62 (83 %)	2.2 min.
			6.27 (17 %)	
	^{207}Tl	Beta		4.8 min.
^{228}Ra		Beta		5.77 years
	^{224}Ac	Beta		6.13 hours
^{224}Ra		Alpha	5.68 (95 %)	3.6 days
			5.44 (5 %)	
	^{220}Rn	Alpha	6.28	54 s.
	^{216}Po	Alpha	6.78	0.16 s.
	^{212}Pb	Beta		10.6 hours
	^{212}Bi	Beta (64 %)		60 min.
		Alpha(36 %)	6.09 (27 %)	
			6.05 (70 %)	
			Others	
	^{212}Po	Alpha	8.78	0.3 us.
	^{208}Tl	Beta		3.1 min.

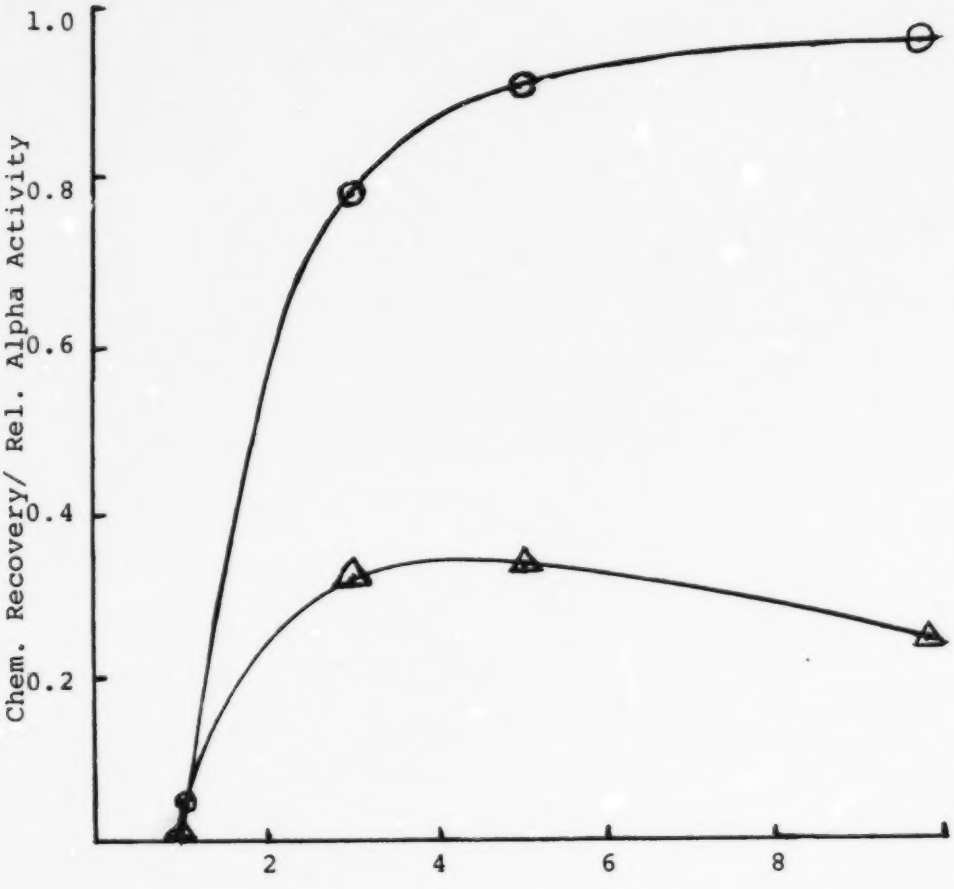

Figure 1. Effect of Added $BaSO_4$ Suspension on Chemical Recovery (⊙) and Relative Alpha Activity (△).

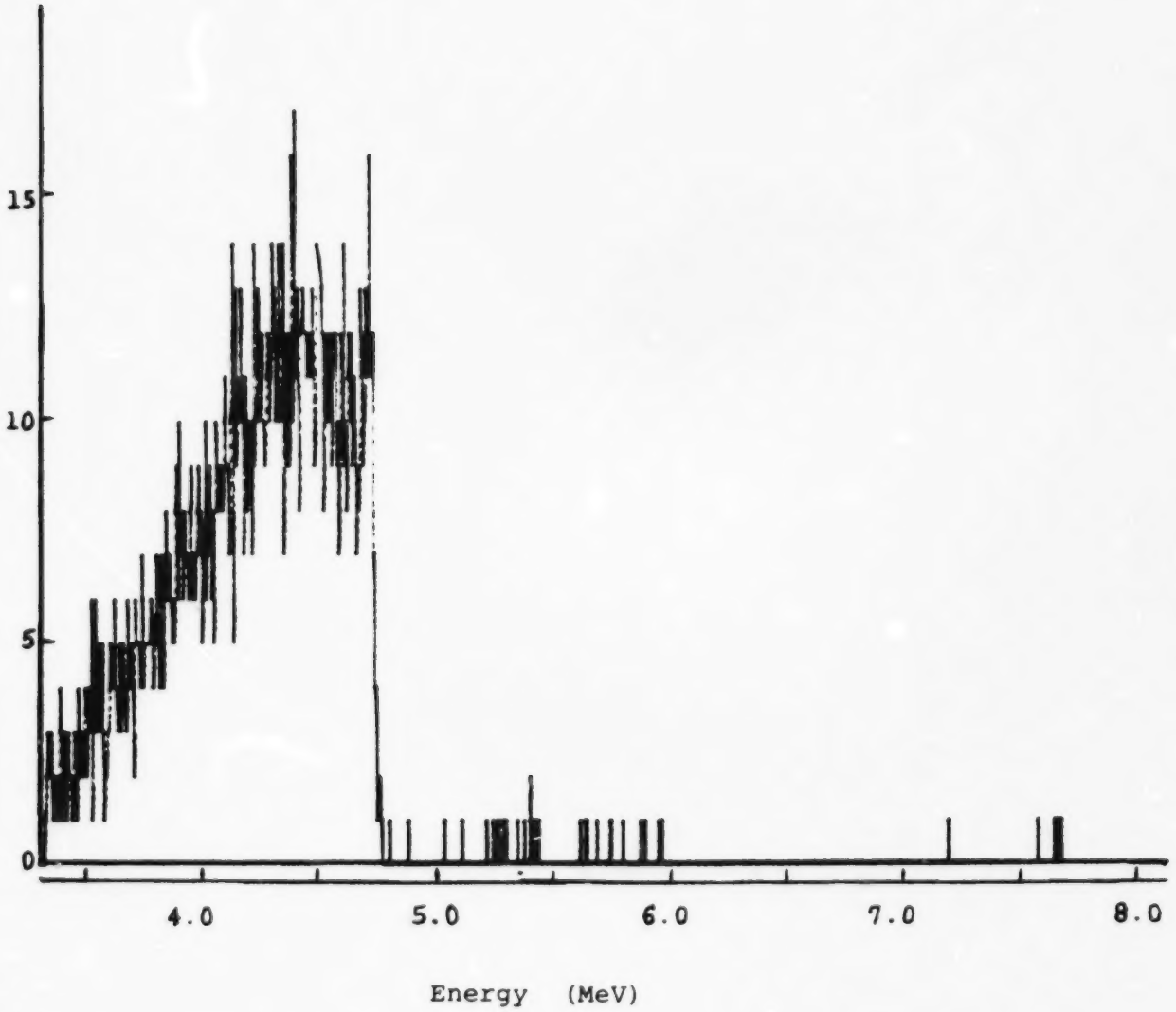

Figure 2. Alpha Spectrum of a Freshly Separated Radium Sample.

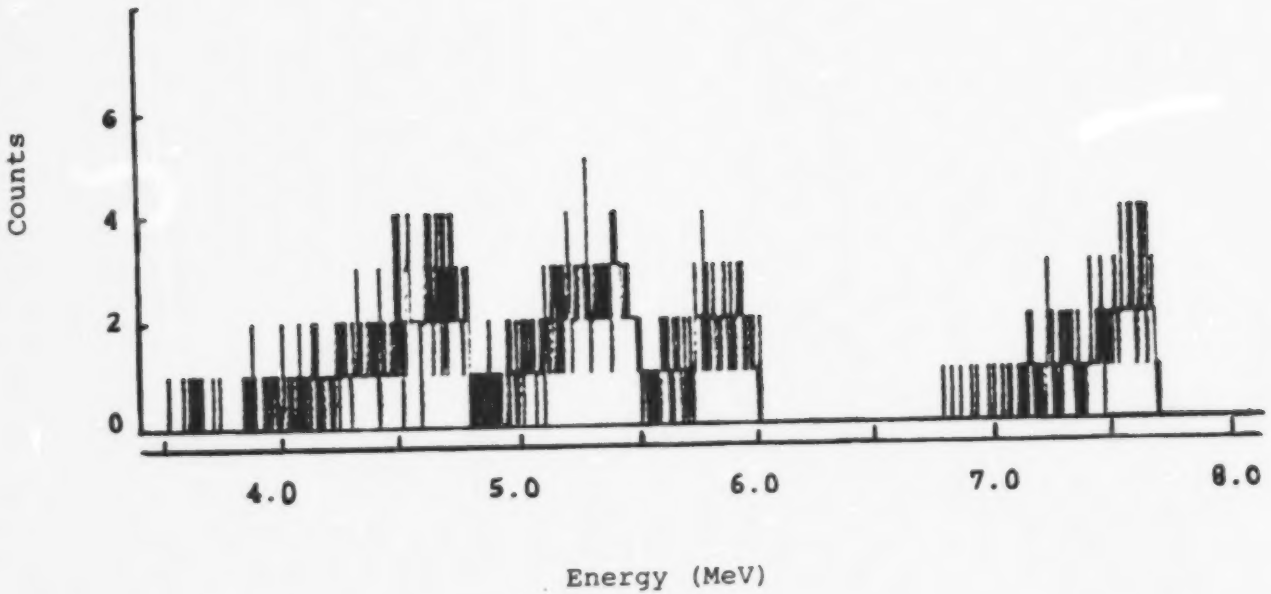

Figure 3. Alpha Spectrum of a 10 Days Old Radium Sample.

APPENDIX

ANALYTICAL PROCEDURE

DETERMINATION OF RADIUM-226 IN GROUND WATER SAMPLES

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DETERMINATION OF RADIUM-226 IN GROUND WATER SAMPLES

EQUIPMENT AND SUPPLIES

- A clean laboratory working area containing a fume hood and an analytical balance.
- Various reagents and chemicals as detailed in a separate section entitled " REAGENTS AND SOLUTIONS ".
- Hot plate stirrer, vacuum filtration equipment, glassware and other laboratory ware as described in a separate section entitled " APPARATUS ".
- EG&G ORTEC Model 576 alpha spectrometer with 450 mm^2 silicon surface barrier detectors coupled to an EG&G ORTEC 7052 data acquisition and analysis system.
- A 76 mm X 76 mm NaI(Tl) detector coupled to a Canberra Series 30 multichannel analyzer.

STANDARDIZATION

- Calibrate and set the region of integration for Ra-226.
- Determine the counting efficiency of the alpha spectrometer for Radium-226 using the same source geometry as the sample.
- Determine the reagent blank count rate for Radium-226.
- Calibrate and set the window for counting Barium-133 tracer in the gamma counting system and determine the background count rate in the region of interest.

1. OUTLINE OF THE METHOD

Radium in the sample is co-precipitated with barium as sulphates on a specially prepared barium sulphate suspension. The presence of complexing agents is used to prevent the precipitation of other alpha emitting radionuclides. The precipitate is filtered through a Millipore filter and counted for barium-133 to determine the chemical yield and in the alpha spectrometer to determine the radium-226 activity

2. INTERFERENCE

The procedure is designed to remove interferences due to other alpha emitting radionuclides. Interference from other alpha emitting radium isotopes and decay products of all radium isotopes is reduced by restricting the integration of the counts to between 3.4 to 4.8 Mev in the alpha spectrum.

3. CONCENTRATION RANGE

The lower limit of detection depends on the reagent blank and the counting period. For a counting time of 1000 minutes and the present clean detector system the minimum detectable activity is about 0.2 pCi. An upper limit of 100 pCi has been arbitrarily set to limit the build up of radon contamination in the detector.

4. PRECISION AND ACCURACY

For environmental samples the precision of the method is controlled by the counting statistics. For samples containing >10 pCi of radium a precision of +/- 10 % has been calculated for a 95 % confidence level. The accuracy of the procedure was determined by analyzing an EPA supplied solution of known radium-226 content. The measured value compared well with the known value.

5. REFERENCE

5.1 C. W. Sill; (1980) Analytical Chemistry 52, pp.1452-1459.

6. REAGENTS AND SOLUTIONS

Reagent grade chemicals and distilled water should be used through out the procedure.

6.1	Citric acid, ≈1 mol/L	Dissolve 1 formula weight of citric acid in a liter of water.
6.2	Saturated sodium sulphate solution.	Dissolve sodium sulphate in water to form saturated solution.
6.3	Lead carrier solution, 1 µg Pb/mL	Dissolve 1.6 mg of lead nitrate and dilute to 1 liter with 5 % nitric acid.
6.4	Bismuth carrier solution, 1 µg Bi/mL	Dissolve 2.3 mg of bismuth nitrate and dilute to 1 liter with 5 % nitric acid.
6.5	Ethylenediaminetetra-acetic acid (EDTA) disodium salt, 0.25 mol/L	Dissolve 85 g of disodium EDTA in water and dilute to a liter.
6.6	Barium carrier solution, 0,5 mg Ba /mL	Dissolve 0,9 g of barium nitrate in water and dilute to a liter.
6.7	Barium sulphate suspension	See the note below.
6.8	Hydrochloric acid, 36 % by weight	
6.9	Nitric acid, 71 % by weight	
6.10	Nitric acid, 1:1 by volume	Add 500 mL of 71 % nitric acid to 500 mL of water.
6.11	Ammonium hydroxide, 28 % by weight	
6.12	Barium-133 tracer solution	Dilute 6 µCi of Ba-133 to 100 mL with 5 mol/L HCl

NOTE:

Preparation of Barium Sulphate Suspension

Approximately 0.5 g of barium nitrate is dissolved in 60 mL of distilled water in a 250 mL Erlenmeyer flask. Then 24 g of anhydrous potassium sulphate and 13 mL of sulphuric acid (98 % by weight) are added and the resultant suspension is evaporated until the barium sulphate formed redissolves. The flask is then cooled and the residual cake is slurried in 150 mL of distilled water with heating. The suspension now formed is cooled and its volume made up to 200 mL. The barium sulphate precipitate is resuspended by shaking before use. The suspension is stable and can be used for up to two months. Preparation of a new suspension is recommended if the available suspension is older than 2 months or its age is unknown.

7. APPARATUS

7.1	Pyrex beakers	1500 mL capacity
7.2	Hot plate stirrer	Fisher 11-496-14
7.3	Vacuum pump	Fisher 01-0933-5
7.4	Filtration apparatus	Millipore Corp. Ltd.
7.5	Millipore filters	HAWP 047 00 and HABP 047 00 Millipore Corp. Ltd.
7.6	Oxford pipette	5 and 10 mL capacity
7.7	Eppendorf pipette	0.1 and 1 mL capacity
7.8	Heat lamp	
7.9	pH meter	
7.10	Assorted laboratory ware	Stirring bars, plastic petri dish, etc.

8. SAMPLE PREPARATION

The sample should be clean and free of suspended material. If not, filter it through a Millipore HAWP-047-00 filter into a clean filtering flask. Use one liter of this filtered solution for analysis.

9. PROCEDURE

9.1 To a liter of the sample (or a known volume if 1 liter of the sample is not available), add in order while mixing 10 mL citric acid, 5 mL EDTA solution, 1 mL lead carrier solution, 1 mL bismuth carrier solution, 0.1 mL barium carrier solution, 0.1 mL barium-133 tracer solution, 5 mL barium sulphate suspension and 10 mL saturated sodium sulphate solution.

9.2 Heat the solution to a gentle boil and then reduce the heat and digest the precipitate at approximately 90° C for one hour. Cool the solution and filter it under low vacuum through a Millipore filter HABP-047-00. Scrub the beaker with a rubber policeman and transfer the precipitate quantitatively to the filtering funnel. Care must be taken to reduce the loss of the precipitate to the funnel wall by filtering slowly and steadily such that a minimum of solution is present in the funnel at any time. Wash the precipitate on the filter with 5 mL of 1:1 nitric acid followed by ten 5 mL washings of distilled water. Transfer the filter disc to a plastic petridish and dry it under a heat lamp.

9.3 Count the filter disc for Ba-133 on a NaI(Tl) detector. Count a standard source of 0.1 mL of the tracer in the same geometry and multichannel analyzer settings. Measure the background counts also.

9.4 Remove the unexposed filter disc portion by carefully cutting with a sharp blade. Mount the filter disc in the sample holder of the alpha spectrometer. Place the sample in the detector assembly and evacuate. When the pressure in the chamber reaches < 0.8 mbar turn on the detector bias voltage and wait for a minute. Set the data acquisition and analysis system to the acquiring mode and count the sample for 1000 minutes. Record the integrated Ra-226 counts.

10. DETERMINATION OF COUNTING EFFICIENCY FOR RA-226

The counting efficiency for radium-226 on both the surface barrier detectors should be determined by using a radium solution of known concentration. The procedure is as follows:

> To liter samples of distilled water acidified with 5 mL of 38 % HCl add aliquots of standard radium solution containing approximately 100 pCi of radium-226. Precipitate the sulphates of barium and radium as described in section 9. Run two reagent blanks as above but without any radium in them. Determine the chemical yield for each of these precipitations by counting the barium-133 activities and the activity of the standard barium-133 source as described in section 9.3.

11. CALCULATIONS

11.1 Counting Efficiency for Radium-226

11.1.1 Determine the counts per minute (cpm) recorded for the standard samples.

$$cpm(std) = \frac{\text{Gross integrated counts for radium-226}}{\text{Time in minutes}}$$

11.1.2 Determine the cpm recorded for the blank.

$$cpm(blk) = \frac{\text{Gross integrated counts for radium-226}}{\text{Time in minutes}}$$

11.1.3 Determine the chemical yield of each source.

$$\text{Chem. yield} = \frac{\text{Ba-133 (cpm(sample) - cpm(bkg))}}{\text{Ba-133(cpm(std. source) - cpm(bkg))}}$$

11.1.4 Correct the cpm for chemical yield.

$$cpm(cor.) = cpm \;/\; \text{Chem. yield}$$

11.1.5 Determine the net cpm for the standard.

$$\text{Net } cpm(std,cor.) = cpm(std,cor.) - cpm(blk,cor.)$$

11.1.6 Determine the counting efficiency.

$$E(Ra\text{-}226) = \frac{\text{Net cpm(std,cor.)}}{\text{dpm of Ra-226 taken}}$$

11.2 Calculation of Radium-226 Activity in the Sample

11.2.1 Determine the net corrected cpm of the sample .

$$\text{cpm(spl)} = \frac{\text{Gross integrated counts for radium-226}}{\text{Time in minutes}}$$

cpm(spl,cor.) = cpm(spl) / Chem. yield

Net cpm(spl,cor.) = cpm(spl,cor.) - cpm(blk,cor.)

11.2.2 Determine the disintegration per minute (dpm) of the sample radium-226 content.

dpm(spl) = Net cpm(spl,cor.) / E(Ra-226)

11.2.3 Determine the activity of the sample.

$$\text{Activity} = \frac{\text{dpm(spl)}}{\text{2.22 x Vol. of the sample}} \quad \text{pCi/L}$$

11.3 Calculation of the Standard Deviation of the Measurements

11.3.1 Determine the uncertainty in the cpm (S (cpm)).

$$S\ (cpm) = (cpm(spl)/t(spl) + cpm(blk)/t(blk))^{1/2}$$

11.3.2 Determine the uncertainty in the dpm (S (dpm)).

$$S\ (dpm) = \frac{S\ (cpm)}{\text{Chem. yield X E(Ra-226)}}$$

11.3.3 Determine the standard deviation of the measurement.

$$\text{Std. Dev.} = S(\text{dpm}) \times \left(\left(S(\text{dpm})/\text{dpm}\right)^2 + \left(S(E(\text{Ra-226}))/E(\text{Ra-226})\right)^2 + \left(S(\text{Chem. yield})/\text{Chem. yield}\right)^2 \right)^{1/2}$$

11.3.4 The percent relative standard deviation can then be calculated.

$$\%\ \text{Rel. Std. Dev.} = \frac{\text{Std. Dev.} \times 100}{\text{dpm}}$$